Flashpoint Using the Pensky-Martens Closed-cup Method

Scope

Method 1010 uses the Pensky-Martens closed-cup tester to determine the flash point of fuel oils, lube oils, suspensions of solids, liquids that tend to form a surface film under test conditions and other liquids.

Summary

The sample is heated at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame ignites the vapor above the sample.

Apparatus and Materials

A. Pensky-Martens Closed Flash Tester, as described in Annex A1 of ASTM Method D93-77. (Automatic flash point testers are available and may be advantageous since they save testing time, permit the use of smaller samples, and exhibit other advantages. If automatic testers are used, the user must be sure to follow all manufacturer's instructions for calibrating, adjusting, and operating the instrument. In any cases of dispute, the flash point as determined manually shall be considered the referee test.)

B. Thermometers: Two standard thermometers shall be used with the ASTM Pensky-Martens tester.

1. For tests in which the indicated reading falls within - 7°C to +110°C (20°F to 230°F), inclusive: either (1) an ASTM Pensky-Martens Low Range or Tag Closed Tester Thermometer having a range from -7°C to +110°C (20°F to 230°F) and conforming to the requirements for thermometers 9C (9F) and as prescribed in ASTM Specification E1, or (2) an IP Thermometer 15 C (15F) conforming to the specifications given in Annex A3 of ASTM D93-77.

2. For tests in which the indicated reading falls within 110°C to 370°C (230°F to 700°F): either (1) an ASTM Pensky-Martens High Range Thermometer having a range from 90°C to 370°C (200°F to 700°F) and conforming to the requirements for Thermometers 10C (10F) as prescribed in Specifications E1, or (2) IP Thermometer 16C (16F) conforming to specifications given in Annex A3 of ASTM D93-77.

Reagents

A. Calcium Chloride.

B. p-Xylene reference standard.

Sample Collection, Preservation, and Handling

A. All samples must be collected using a sampling plan that addresses the considerations discussed in section 1 of Test Methods for Evaluating Solid Waste, Physical/Chemical Methods USEPA, July, 1982, Method 1010.

B. Samples shall not be stored in plastic bottles since volatile materials may diffuse through the walls of the bottle.

Procedure

A. Preparation of samples: Samples that do not contain volatile contaminants shall be prepared in the following manner. If the sample is suspected of containing volatile contaminants, the treatments described in steps B and C should be omitted.

B. Samples of very viscous materials may be warmed until they are reasonably fluid before they are tested. However, no sample should be heated more than is absolutely necessary and no sample should be heated to a temperature that exceeds 17°C (30°F) below the sample's expected flash point.

C. Samples containing dissolved or free water may be dehydrated with calcium chloride or by filtering through a qualitative filter paper or a loose plug of dry absorbent cotton. Warming the sample is permitted but it shall not be heated for

prolonged periods or above a temperature of 17°C (30°F) below the sample's flash point.

D. Thoroughly clean and dry all parts of the cup and its accessories before starting the test. Be sure to remove any solvent that was used to clean the apparatus. Fill the cup with the sample to be tested to the level indicated by the filling mark. Place the lid on the cup and set the latter in the stove. Be sure to properly engage the locating or locking device. Insert the thermometer. Light the test flame and adjust it to a diameter of 5/32 in (4mm). Supply the heat at such a rate that the temperature as indicated by the thermometer increases 5°C to 6°C (9°F to 11°F)/minute. Turn the stirrer at 90 to 120 rpm, stirring in a downward direction.

E. If the sample is expected to have a flash point of 110°C (230 °F) or below, apply the test flame when the temperature of the sample is from 17°C (30°F) to 28°C (50°F) below the expected flash point and thereafter at a temperature reading that is a multiple of 1°C (2°F). Apply the test flame by operating the mechanism on the cover which controls the shutter and test flame burner so that the flame is lowered into the vapor space of the cup in 0.5 seconds, left in its lowered position for 1 second and quickly raised to its high position. Do not stir the sample while applying the test flame.

F. If the sample is expected to have a flash point above 110°C (230°F), apply the test flame in the manner just described at each temperature that is a multiple of 2°C (5°F), beginning at a temperature of 17°C (30°F) to 28°C (50°F) below the expected flash point. When testing materials to determine if volatile contaminants are present it is not necessary to adhere to the temperature limits for initial flame application as stated in B and C.

G. Record as the flash point the temperature read on the thermometer at the time the test flame application causes a distinct flash in the interior of the cup. Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame at applications preceding the one that causes the actual flash. The actual flash will have occurred when a large flame propagates itself over the surface of the sample. If the temperature reaches 150°F without a flash point, report the value as >150°F. Samples are considered hazardous if the flash point is 140°F or less.

H. Determination of the flash point of suspensions of solids and highly viscous materials.

1. Bring the material to be tested and the tester to a temperature of 15±5°C

($60\pm10^{o}F$) lower than the estimated flash point. Turn the stirrer at 250 ± 10 rpm, stirring in a downward direction. Raise the temperature throughout the duration of the test at a rate of not less than $1^{o}C$ nor more than $1.5^{o}C$ (2 to $3^{o}F$)/minute. With the exception of these requirements for rates of stirring and heating proceed as described previously.

Calculation

Observe and record the ambient barometric pressure at the time of the test. When the pressure differs from 760 mm Hg (101.3 kPa), correct the flash point as follows:

(A) Corrected flash point = C + 0.25 (101.3-P)
(B) Corrected flash point = F + 0.06 (760-P)
(C) Corrected flash point = C + 0.033 (760-P)

Where: F = observed flash point, ^{o}F.
C = observed flash point, ^{o}C.
P = ambient barometric pressure, mm Hg.
p = ambient barometric pressure, kPa.

NOTE: The barometric pressure used in this calculation must be the ambient pressure for the laboratory at the time of test. Many aneroid barometers, such as those used at weather stations and airports, are precorrected to give sea level readings. They must not be used.

Record the corrected flash point to the nearest $0.5^{o}C$ or $1^{o}F$.

Report the recorded flash point as the Pensky-Martens Closed Cup Flash Point, ASTM D93-IP 34, of the sample tested.

Refer to method ASTM D93 77 for more details and background on the Pensky-Marten method.

Quality Control

The flash point of the p-Xylene reference standard must be determined in duplicate at least once per sample batch. The average of the two analyses should be $27\pm0.8^{o}C$ ($81\pm1.5^{o}F$).

Bibliography

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods USEPA, July 1982, 2nd Edition, Method 1010.